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Indian Standard
SPECIFICATION FOR ETHYLENE DICHLORIDE-CARBON TETRACHLORIDE MIXTURE (3 : 1 *v/v*)

(Revised)

UDC 615·778 : 547·413·1 + 547·412·133

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

Price Rs 4·50 ~~Gr 5~~ Gr 6 *October* 1965

Indian Standard

SPECIFICATION FOR ETHYLENE DICHLORIDE-CARBON TETRACHLORIDE MIXTURE ($3:1\,v/v$)

(Revised)

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Indian Standard

SPECIFICATION FOR ETHYLENE DICHLORIDE-CARBON TETRACHLORIDE MIXTURE (3 : 1 *v/v*)

(*Revised*)

0. FOREWORD

0.1 This Indian Standard (Revised) was adopted by the Indian Standards Institution on 18 September 1965, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Ethylene dichloride-carbon tetrachloride mixture (EDCT) (see 2.1) in the proportion of 3 : 1 (*v/v*) is very extensively used in India for the fumigation of food grain and other products. Ethylene dichloride by itself is a powerful fumigant for pests of stored food grain. Carbon tetrachloride too is a fumigant, but it is added to ethylene dichloride primarily to reduce the fire risk of the latter. Because of the inflammable nature of ethylene dichloride, purchasers in India do not procure the two components separately for mixing them under their own charge and instead ask for only the ready-made mixture which is available in the country under different proprietary names.

0.3 No detailed specification for the ready-made mixture has been so far laid down by any organization in the world, although specifications for the two components do exist. At the request of trade organizations and with the support of government departments concerned, the Indian Standards Institution has formulated this standard, because separate specifications for ethylene dichloride and carbon tetrachloride do not help the consumer in judging the quality of the mixture.

0.4 This standard is based on actual physical and chemical properties found out for a number of samples of the material These samples were obtained from Excel Industries Ltd., Bombay, and were analysed in the laboratories of Tata Chemicals Ltd., Mithapur; Burmah-Shell Oil Storage and Distributing Co. of India Ltd., Bombay; Excel Industries Ltd., Bombay; and Shri Ram Institute for Industrial Research, Delhi.

0.5 This standard was first published in 1957. In that standard, it was specified that a minimum of 97 percent of EDCT shall distil within the range of 75” to 83°C at 760 mm pressure. M/s Chika Private Limited, Bombay, had sometime back raised the question that distillation range as specified in the Indian Standard was not correct as compared to the analytical report by M/s Chemische Werke Huels, West Germany. The National Chemical Laboratory, Poona, which has also been examining the ethylene dichloride, technical, required in the production of EDCT had also raised the same point that the distillation range as specified in the Indian Standard was very strict. In view of the above, the Sectional Committee decided that a collaborative study should be carried out on the problem by M/s Tata Chemical Limited, Mithapur; M/s Burmah-Shell Oil Storage and Distributing Co. of India Ltd., Bombay; Shri Ram Institute for Industrial Research, Delhi; and Excel Industries Ltd., Bombay; on a number of samples of the mixture. This collaborative work showed that since the two liquids ethylene dichloride and carbon tetrachloride did not form an azeotropic mixture, the components more or less boiled independently and. that only ethylene dichloride was distilling at the last stage. It was, therefore, decided to specify the maximum temperature in the range as it applied to ethylene dichloride and as specified in the standard for ethylene dichloride and also to feduce the volume of the mixture that should distil over within the specified range from 97 percent to 95 percent. Some changes regarding the requirement for residue on evaporation and acidity had also to be effected in the standard apart from the revision of the method of sampling taking into account the statistical principles of sampling. In view of the above, the Sectional Committee decided to issue the revised version of the standard incorporating all the above changes and also the amendment regarding the packing of the material which was issued to it in May 1964.

0.6 This standard is one of a series of Indian Standards on pesticides and their formulations.

0.7 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of test for the ready-made mixture of ethylene dichloride - carbon tetrachloride (ethylene dichloride 75 parts by volume and carbon tetrachloride 25 parts by volume), intended for use as a fumigant.

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*Rules for rounding off numerical values (*tevised).*

2. ABBREVIATION

2.1 For the purpose of this standard, the abbreviation EDCT shall mean the mixture of ethylene dichloride carbon and tetrachloride in the proportion of 3 : 1 (*v/v*).

3. REQUIREMENTS

3.1 Description — The material shall be obtained by mixing 75 parts by volume of ethylene dichloride (see IS : 869-1956*) and 25 parts by volume of carbon tetrachloride (see IS : 718-1955†). The mixture, so obtained, shall be clear, free from sediment, foreign matter and adulterants. It shall have a characteristic odour resembling that of chloroform.

3.2 **Colour** -The colour of the material, when tested in a colorimeter, shall not be darker than a freshly prepared solution of 0·003 0 g of potassium dichromate ($K_2Cr_2O_7$) in 1 000 ml of water,

3.3 The material shall also comply with the requirements specified in Table 1.

TABLE 1 REQUIREMENTS FOR EDCT

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix	Method in IS : 1448 (Part I)-1960*
(1)	(2)	(3)	(4)	(5)
i)	Specific gravity at 27°/27°C	1·316 to 1·336	A	—
ii)	Refractive index at 27°C	1.443 3 to 1·445 3	B	—
iii)	Residue on evaporation, mg/100 ml, *Max*	10·0	C	—
iv)	Distillation range at 75” to 86°C at 760 mm pressure, percent by volume, *Min* (*see* Note j	95	D	—
v)	Chlorine content, percent by weight	76·7 to 78.9	E	—
vi)	Acidity (as HCl), percent by weight, *Mm*	0·002	F	—
vii)	Flash point (Abel)	Not below 49°C	—	P : 20

NOTE -The distillation of the mixture will commence not below 75°C and 95 percent of the material would have distilled over by 86°C.

*Methods of test for petroleum and its products, Part I.

---

*Specification for ethylene dichloride, technical.

†Specification for carbon tetrachloride, technical.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in clean and dry containers made of galvanized iron or mild steel drums. For smaller packs of less than one kilogram, glass containers, suitably protected, may be used.

4.2 Marking—The following information shall be marked legibly and indelibly on each container:

a) Common name of the material,

b) Name of the manufacturer,

c) Net weight of the material,

d) Batch number, and

e) The minimum cautionary notice worded as under:

> HAZARDOUS. PREVENT INHALATION OF FUMES, AND CONTAMINATION OF SKIN. USE RUBBER GLOVES, OVERALLS AND RESPIRATOR WHEN FUMIGATING. STORE AWAY FROM FOODSTUFF AND ANIMAL FEED. DO NOT USE CONTAINER FOR STORAGE OF FOODSTUFF AND ANIMAL FEED. DO NOT SMOKE WHEN WORKING WITH THIS MATERIAL AND WASH HANDS THOROUGHLY WITH SOAP AND PLENTY OF WATER AFTER HANDLING.

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE—The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix G.

6. TESTS

6.1 Tests shall be carried out as prescribed in 3.1 and 3.2 and the appropriate appendices given under col 4 and 5 of Table 1.

6.2 **Quality of Reagents —** Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1960*) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Specification for water, distilled quality (*revised*).

APPENDIX A

[*Table* 1, Item (i)]

DETERMINATION OF SPECIFIC GRAVITY

A-1. TERMINOLOGY

A-1.1 For the purpose of this standard the specific gravity of the material shall be the ratio of the weight in air of a given volume of the material at 27°C to that of an equal volume of water at the same temperature.

A-2. METHOD

A-2.1 The specific gravity shall be determined with a specific gravity bottle of Castell Evans modification with ground-in stopper and ground-on cap (Fig. 1) or with a pyknometer tube of the Perkin's type (Fig. 2).

A-3. PROCEDURE

A-3.1 Weigh the clean, dry, specific gravity bottle or pyknometer of 25 ml capacity. Fill the tared specific gravity bottle or pyknometer with freshly boiled, distilled water and place it in a bath maintained at 27” ± 1°C and allow sufficient time to attain the temperature of the bath. Then insert the ground-in stopper which has also been brought to 27” ± 1°C. Wipe the excess liquid from the stopper and place the cap on it, in position, remove the specific gravity bottle or the pyknometer from the bath, bring to room temperature and weigh. Empty the specific gravity bottle or the pyknometer, clean and dry it, and repeat the operation with the material at 27” ± 1°C.

A-4. CALCULATION

A-4.1 Calculate the specific gravity as follows:

$$\text{Specific gravity at } 27°/27°\text{C} = \frac{C - A}{B - A}$$

where,

C = weight in g of the specific gravity bottle or pyknometer filled with the material at 27°C,

A = weight in g of the dry specific gravity bottle or pyknometer, and

B = weight in g of the specific gravity bottle or pyknometer filled with water at 27°C.

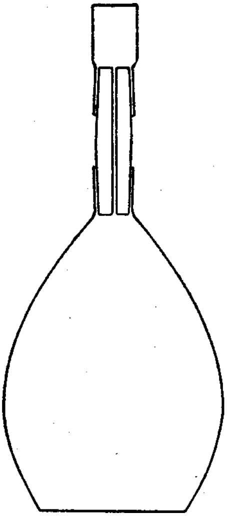


FIG. 1 SPECIFIC GRAVITY BOTTLE (CASTELL EVANS MODIFICATION WITH GROUND-IN STOPPER AND GROUND-ON GAP)

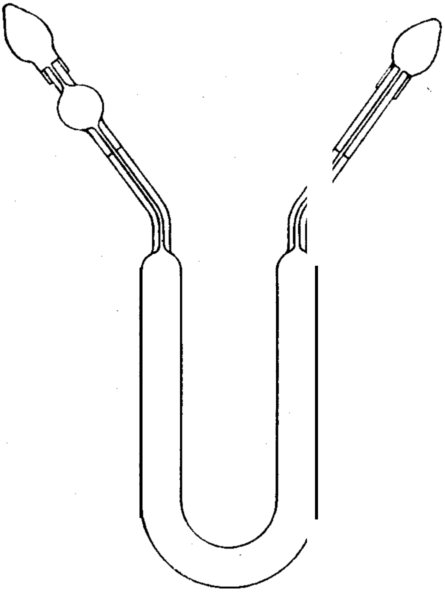


FIG. 2 PYKNOMETER TUBE (PERKIN'S TYPE)

APPENDIX B

[*Table* 1, Item (ii)]

DETERMINATION OF REFRACTIVE INDEX

B-1. APPARATUS

B-1.1 Refractometer — Abbe refractometer is recommended. The temperature of the refractometer should be controlled to within ±0·1°C, and for this purpose it should be provided with a thermostatically controlled water-bath and a motor-driven pump to circulate water through the instrument. The instrument should be standardized, following the manufacturer's instructions, with a liquid of known purity and refractive index or with a glass prism of known refractive index. Distilled water is a satisfactory liquid for standardization.

B-1.2 Light Source — If the refractometer is equipped with a compensator, a tungsten lamp *or* a daylight bulb may be used. Otherwise, a monochromatic light, such as an electric sodium vapour lamp, shall be used.

B-2. TEST TEMPERATURE

B-2.1 The test shall be carried out at 27·0° ± 0·1°C.

B-3. PROCEDURE

B-3.1 Adjust the temperature of the refractometer to 27·0° ± 0·1°C. Ensure that the prisms are clean and completely dry. Close the prisms by manipulating the screw provided for the purpose. Slacken the screw and pour a few drops of the material into the funnel-shaped aperture of a narrow passage provided in the prism casing. Tighten the screw so that the material is distributed by capillary action over the entire space between the two prisms. Allow to stand for one or two minutes. Adjust the instrument and light to obtain the most distinct reading possible, and determine the refractive index. Take a second reading after a lapse of few minutes.

B-3.1.1 *Precaution* — Since moisture in. the air may condense on the cooled prisms, great care should be taken when determinations are made in hot and humid weather, when the ambient temperature exceeds 27°C.

B-3.1.2 *Temperature Correction* — If, for any reason, it is not possible to determine the refractive index at the specified test temperature, approximate corrections shall be made using the following formula:

$$R = R' + 0{\cdot}000\,45\,(T' - T)$$

where

R = the reading of the refractometer reduced to the specified test temperature, that is 27°C,

R' = the reading of the refractometer at T'°C,

T' = the temperature at which the reading R' is taken, and

T = the specified test temperature, that is 27°C.

APPENDIX C

[*Table* 1, Item (iii)]

DETERMINATION OF RESIDUE ON EVAPORATION

C-1. PROCEDURE

C-1.1 Evaporate 100 ml of the material, accurately measured in a tared nickel, silica, porcelain, resistance glass or platinum basin of about 75 mm diameter and 200 mm deep, on a water-bath. When all the material has evaporated heat the basin for one hour at 110° ± 2°C in an air-oven. Cool it in a desiccator and weigh. Record the weight. The difference in the two weights represents the residue on evaporation.

APPENDIX D

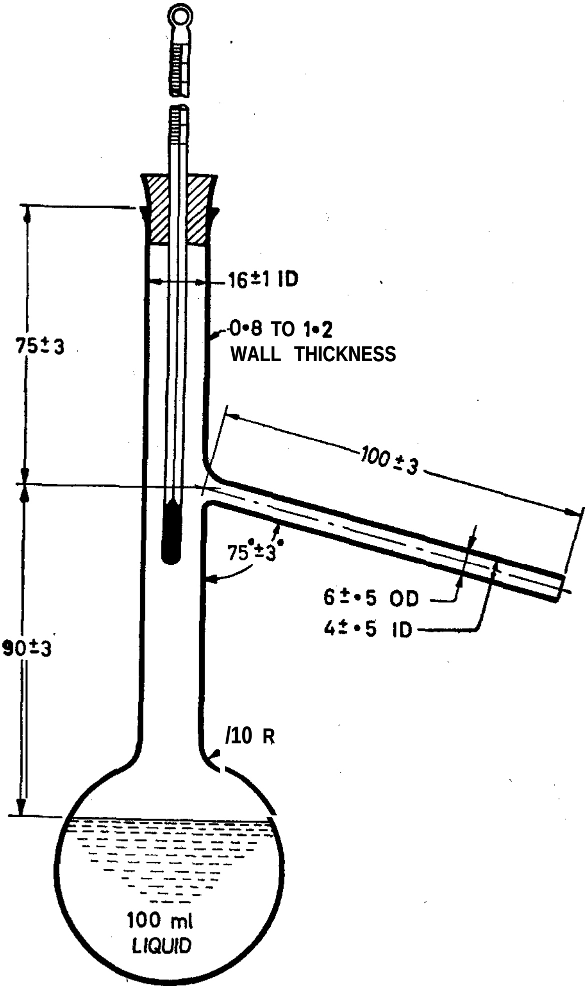
[*Table* 1, Item (iv)]

DETERMINATION OF DISTILLATION RANGE

D-1. APPARATUS

D-1.1 Distillation Flask — of the shape and dimensions given in Fig. 3. Fix the flask in the vertical position by means of a clamp at the extreme upper end of the neck.

D-1.2 Thermometer — so fitted in the flask that the bottom of the capillary is at level with the lower edge of the side tube joint and the immersion mark is level with the top of the work.



All dimensions in *millimetres.*

FIG. 3 DISTILLATION FLASK (CAPACITY 130 ± 5 ml)

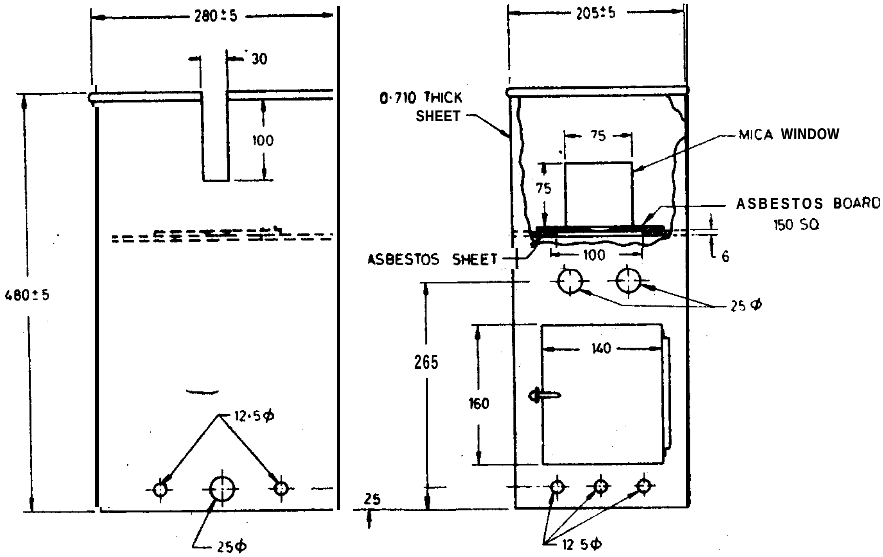
D-1.2.1 The recommended dimensions, tolerances and graduations of the thermometer shall be as follows:

Range	48° to 102°C
Graduation	0·2°C
Longer lines at each	1°C
Fully figured at each	10°C
Fractional figuring at each	2°C
Immersion	*100* mm
Overall length	380 to 385 mm
Length of main scale, *Min*	190 mm
Bulb length	15 to 20 mm
Stem diameter	6·0 to 7·5 mm
Distance from bottom of the bulb to bottom of main scale	125 to 145 mm
Distance from bottom of the bulb to top of contraction chamber, *Mux*	35 mm
Maximum error	0·2°C
Maximum error in an interval	0·2°/10°C

D-1.2.2 The thermometer shall bear a certificate of the National Physical Laboratory, New Delhi, or any other institution authorized by the Government of India to issue such a certificate.

D-1.3 Rectangular Draught Screen — made of 0·7 10 mm thick sheet metal, with the dimensions shown in Fig. 4 and open at the top and bottom. It shall comply with the following requirements:

a) In each of the two narrow sides of the draught screen there shall be two circular holes, each 25 mm in diameter, and in each of the four sides of the draught screen, there shall be three holes with their centres 25 mm above the base of the draught screen. These holes shall occupy the position shown in Fig. 4, the diameter of each of the holes centrally situated in the longer sides shall be 25 mm, and of the remaining ten holes, shall be 12·5 mm. At the middle of each of the wider sides a vertical slot with the dimensions shown in Fig. 4 shall be cut downwards from the top of the screen.



All dimensions in millimetres.

FIG. 4 RECTANGULAR DRAUGHT SCREEN

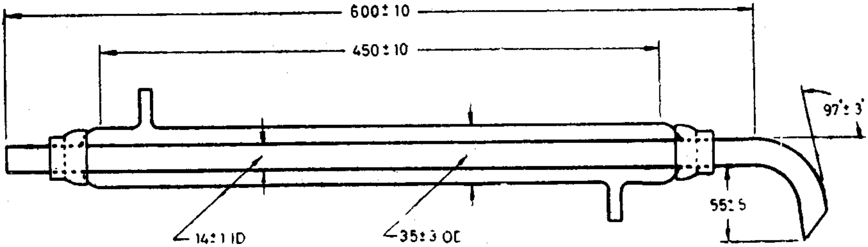
b) A sheet of hard asbestos, 6 mm in thickness, and having a central circular hole, 100 mm in diameter, shall be supported horizontally in the screen and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos sheet may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

c) In one of the narrow sides of the screen a door shall be provided having the dimensions and position as shown in Fig. 4. In each of the narrow sides of the screen a mica window shall be placed centrally, with the bottom of the window on a level with the top of the asbestos sheet. The dimensions and position of the windows are shown in Fig. 4.

d) In addition to the asbestos sheet referred to above, an additional asbestos board 150 mm square is required. The additional asbestos board shall be 6 mm in thickness and shall have a central hole 30 mm in diameter. This shall be placed on the asbestos sheet described above.

D-1.4 **Liebig** Condenser — with the bent and made of good quality resistance glass, with a wall thickness of 1·0 to 1·5 mm and conforming to the shape and dimensions given in Fig. 5.

D-1.4.1 Alternatively, the bent portion may be substituted by an adapter fitted externally in such a manner that the distillate does not come in contact with the cork.



All dimensions in millimetres.

FIG. 5 LIEBIG CONDENSER

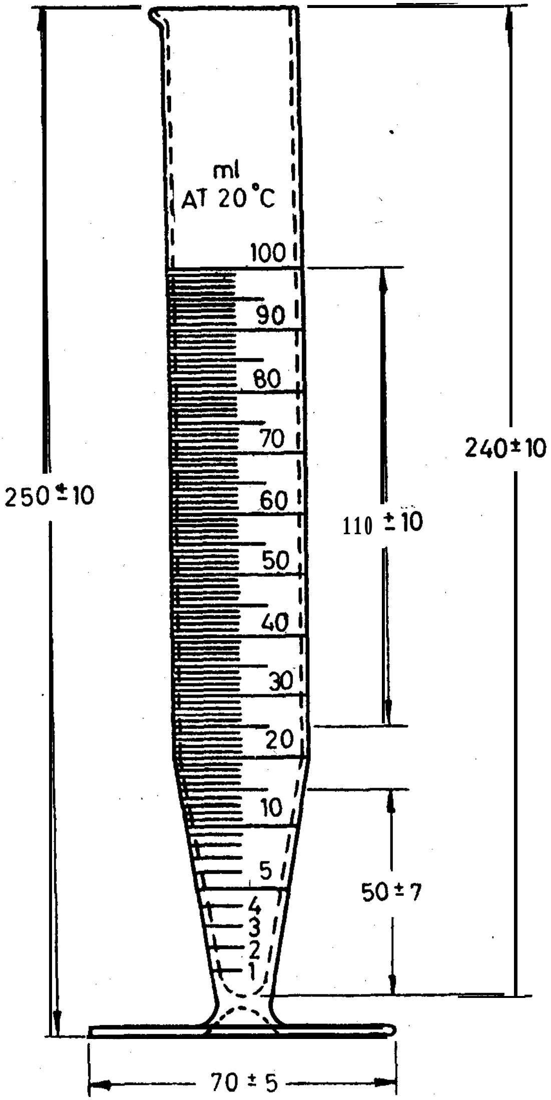
D-1.5 **100-ml Crow Receiver (Fig. 6) or Graduated Cylinder** — with millilitre marks running half-way round the circumference, with 5 millilitre marks running three-quarters-way round and with 10 millilitre marks running all round the circumference, and numbered.

D-1.6 **Bunsen Burner** — giving an entirely non-smoky flame and provided with an adjustable screw-clip with the help of which it is possible to lower or raise the flame according to requirement.

D-1.7 Half-Second Pendulum — for measuring the rate of distillation.

D-2. PROCEDURE

D-2.1 Assemble the apparatus as shown in Fig. 7. Measure 100 ml of the material at the laboratory temperature in the 100-ml crow receiver and transfer it to the distillation flask, the contents of the receiver being allowed to drain for 15 seconds into the flask. Add a fragment (about 2 mm cube) of porous pot or other suitable inert material to prevent bumping, connect the flask to the condenser and insert the thermometer. Pass an adequate supply of cooling water through the condenser. To receive the distillate, use the crow receiver in which the sample was measured, without rinsing or drying. Heat the flask slowly, especially after ebullition has begun, in order that the mercury column of the thermometer may become fully expanded before the first drop of distillate falls into the receiver, care being taken that the total period of this preliminary heating shall be not less than 5 nor greater than 10 minutes. Place the receiver so that the condensate will flow down its side. Continue the distillation at the rate of 4 to 5 ml per minute (about 2 drops per second). Read the volume of distillate in the receiver when the thermometer indicates each of the specified distillation temperatures, the temperatures on the thermometer scale being corrected as specified under D-3. The difference between the volumes so recorded is the percentage by volume distilling between the specified temperatures at 760 mm pressure.



All dimensions in millimetres.

FIG. 6 100-ml CROW RECEIVER

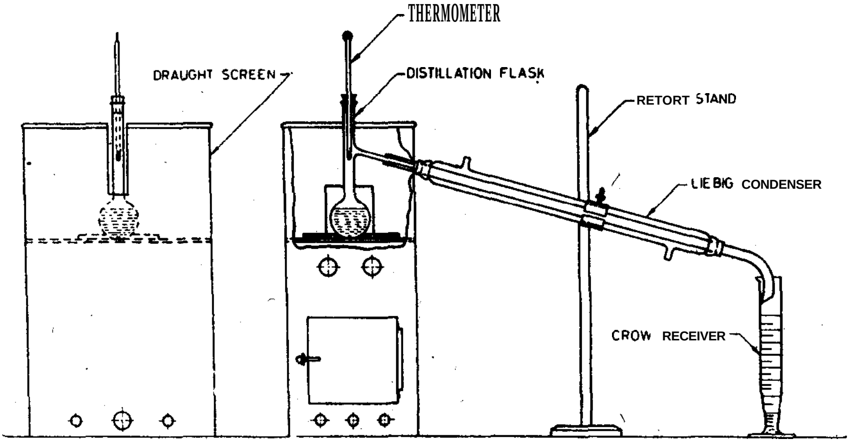


FIG. 7 ASSEMBLY OF APPARATUS

D-3. CORRECTION OF THE THERMOMETER READING

D-3.0 The following corrections shall be applied before starting distillation.

D-3.1 **‘Error of Scale** — In all thermometer readings, make the corrections as indicated on the certificate of the instrument.

D-3.2 **Correction for Barometric Pressure** — If the barometric pressure prevailing during the determination is normal, namely, 760 mm of mercury, no correction need be applied to the specified temperature, and the thermometer scale as corrected under D-3.1 shall be used as such. If, however, the prevailing pressure deviates from 760 mm, the specified temperature shall also be corrected as follows:

a) For every 100 mm above 760 mm, add 0·42°C to the specified temperature, and

b) For every 10 mm below 760 mm, subtract 0·42°C from the specified temperature.

These corrections shall be applied in proportion at the above rate for any prevailing barometric pressure and are valid down to a pressure of 700 mm of mercury.

APPENDIX E

[*Table* 1, *Item* (v)]

DETERMINATION OF CHLORINE CONTENT

E-l. APPARATUS

E-l.1 Tared Small Glass Tube -sealed at one end and drawn to a capillary at the other. The total length of the tube is not less than 4 cm. The size of the tube is such that it could be easily introduced into the bomb tube (E1.2).

E-l.2 Bomb Tube — a thick walled, hard glass tube, about 50 cm long and sealed at one end.

E-l.3 Tube or Bomb Furnace

E-2. REAGENTS

E-2.1 Fuming Nitric Acid

E-2.2 Silver Nitrate — crystals.

E-2.3 Ammonium Hydroxide Solution — sp gr 0·90.

E2.4 Concentrated Nitric Acid — sp gr 1·42 (conforming to analytical reagent grade of IS :264-1950*).

E-3. PROCEDURE

E-3.1 Hold the tared small glass tube by means of a pair of tongs and heat its sealed end to redness on a non-luminous Bunsen flame. Immediately introduce its capillary opening into the material previously taken in a beaker. Allow the tube to cool to room temperature while holding it in that position. (A column of the material would have been drawn into the tube through the capillary opening by the time the tube comes to room temperature.) Take the tube out, invert it and gently tap it on the sides to bring the column of material to the bottom of the tube. Seal the capillary opening in a Bunsen flame. Weigh this filled tube and note the weight of the material contained in it.

E-3.2 Introduce into the bomb tube 3 to 4 g of fuming nitric acid and a slight excess of finely powdered silver nitrate. Take the small glass tube containing the weighed quantity of the material and cut it open at the capillary end. Carefully slip the small glass tube into the bomb tube in such a manner that the contents of the small glass tube do not come into contact with the contents of the bomb tube. Immediately seal the bomb tube without disturbing its contents and carefully anneal it in a luminous flame. Cool the bomb tube, warp in an asbestos paper and place in an inclined position in the tube or bomb furnace. Raise the temperature of the furnace gradually to 300°C or higher, if possible, and keep the bomb tube at that temperature for at least 6 hours. Allow it to cool in the furnace and when cold, open with a Bunsen flame. (The heat would soften the glass and the pressure inside the bomb tube blows open a hole in the softened portion, while the gas would escape.) Cut off the top of the bomb tube with a sharp file.

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*Specification for nitric acid.

E-3.3 Transfer the contents of the bomb tube along with the small glass tube to a glass mortar. Wash out with water the last traces of the precipitated silver chloride from_ the bomb tube into the glass mortar. Break the small glass tube into small pieces by means of a glass pestle taking care that the contents of the mortar do not spill out. Filter the contents of the glass mortar through a quantitative filter paper transferring all the precipitate and the glass pieces on to the filter by washing with water. Wash the filter with-water till the washings are free from traces of silver nitrate. Transfer the filter paper containing the silver chloride and the glass pieces to a beaker. Dissolve the precipitate stirring with an excess of ammonium hydroxide solution and filter the resulting solution quantitatively through a filter paper. Collect the filtrate in another beaker and re-precipitate the silver chloride by adding an excess of nitric acid. Collect the precipitate of silver chloride on a prepared and weighed Gooch crucible, wash the precipitate free from traces of the acid dry at 140° to 150°C, cool in a desiccator and weigh. Repeat the process of drying, cooling and weighing at half-hour intervals until the difference between two successive weighings is less than one milligram. Note the lowest weight.

E-4. CALCULATION

E-4.1 Chlorine content, percent by weight $= \dfrac{24.74\,(a - b)}{W}$

where

a = weight in g of the Gooch crucible with the precipitate of silver chloride (E-3.3),

b = weight in g of the prepared Gooch crucible (E-3.3), and

W = weight in g of the material taken for the test (E-3.1).

APPENDIX F

[*Table* 1, Item (vi)]

DETERMINATION OF ACIDITY

F-1. REAGENTS

F-1.1 Phenolphthalein Indicator Solution — Dissolve 0·5 g of phenolphthalein in 100 ml of rectified spirit (see IS : 323-1959*), 95 percent by volume, and make the solution faintly pink by adding dilute sodium hydroxide solution.

F-1.2 **Standard Sodium Hydroxide Solution** — 0·01 N.

F-2. PROCEDURE

F-2.1 Transfer 100 ml of the material, accurately measured, into a 300 ml glass-stoppered conical flask. Add 100 ml of freshly boiled and cooled water which has been previously neutralized to phenolphthalein indicator solution and shake vigorously. Allow the layers to separate. Separate the aqueous layer, add to it 0·5 ml of the phenolphthalein indicator solution and titrate with the standard sodium hydroxide solution using a microburette.

F-3. CALCULATION

F-3.1 Using the specific gravity as determined in Apppendix A, calculate the acidity (as HCl) as percentage by weight on the basis that 1 ml of normal sodium hydroxide solution is equivalent to 0·003 65 g of hydrochloric acid.

APPENDIX G

(*Clause* 5.1)

SAMPLING OF EDCT

G-1. GENERAL PRECAUTIONS

G-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

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*Specification for rectified spirit (*revised*).

G-1.1 Samples shall not be taken in an exposed place.

G-1.2 The sampling instrument shall be clean and dry when used.

G-1.3 Proper precautions shall be taken while drawing samples since the material is poisonous.

G1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and 'the receptacles for samples from adventitious contamination.

G-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by agitation or any other suitable means so as to bring all portions into uniform distribution.

G-1.6 The samples shall be placed in suitable, clean, dry and air-tight sample receptacles.

G-1.7 The sample receptacles shall be of such a size that they are almost, but not completely, filled by the sample.

G-1.8 Each sample receptacle shall be sealed air-tight after filling and marked with full details of sampling, the date of manufacture, name of the manufacturer, and other particulars of the consignment.

G-1.9 Samples shall be stored'in such a manner that the temperature of the material does not vary unduly from the normal temperature.

G-2. SCALE OF SAMPLING

G2.1 Lot — All the containers in a single consignment of the material drawn from the same batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

G-2.1.1 Sample shall be tested for each lot for ascertaining conformity of the material to the requirements of this specification.

G2.2 The number (n) of containersto be chosen from the lot shall depend on the size of the lot (N) and shall be as given in Table 3.

TABLE.3 NUMBER OF CONTAINERS TO BE CHOSEN FOR SAMPLING

LOT SIZE N (1)	No. OF CONTAINERS TO BE CHOSEN n (2)
3 to 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	7
Over 110	10

G-2.3 These containers shall be chosen at random from the lot and in order to ensure the randomness of selection, some random number table as agreed to between the purchaser and the vendor shall be used. In case, such a table is not available, the following procedure shall be adopted:

Starting from any container in the lot, count them as I, 2, 3,..., etc, up to r, in a systematic manner, where r is equal to the integral part of the value of N/n, N being the total number of containers in the lot and n the number of containers to be chosen (Table 3). Every rth container thus counted shall be separated until the requisite number of containers is obtained from the lot to give samples for test.

G-3. TEST SAMPLES AND REFEREE SAMPLES

G-3.1 Before drawing the test sample, mix thoroughly the contents of each container selected by agitation or any other suitable means so as to bring all portions into uniform distribution. Draw small portions of the material from different parts of each container selected (Table 3). The total quantity of the material drawn from each container shall be not less than 50 g.

G-3.2 Thoroughly mix all the material drawn from the same container. A small but equal quantity shall be taken for each selected container and shall be well-mixed together so as to form a composite sample of not less than 60 g. This composite sample shall be divided into three equal parts one for the purchaser, another for the vendor, and the third for the referee.

G-3.3 The remaining portions of the material from each container (after the quantity needed for the formation ‘of the composite sample has been taken out) shall be divided into three equal parts. These parts shall be immediately transferred to thoroughly dried sample receptacles which are then sealed air-tight and labelled with all the particulars ofsampling given under G-1.8. The material in each such sealed sample receptacle shall constitute a test sample. These individual test samples shall be separated into three identical sets of test samples in such a way that each set has a sample representing each container selected (Table 3). One of these three sets shall be marked for the purchaser, another for the vendor, and the third for the referee.

G-3.4 **Referee Samples** — Referee samples shall consist of a composite test sample (G-3.2) and a set of individual test sample (G-3.3) marked for this purpose and shall bear the seals of the purchaser and the vendor. These shall be kept at a place agreed to between the two.

G-4. TESTING OF SAMPLES

G-4.0 The number of tests mentioned in G-4.1 and G-4.2 shall. be first conducted on the test sample marked for the purchaser. In the contingencies, such as loss, spoilage of spilling, of the purchaser's test samples or if the vendor so desires, the test samples marked for the vendor may be tested. In case of a dispute, the referee test samples shall be tested and the test results obtained on the referee test samples shall be considered as final.

G-4.1 Test for the determination of specific gravity, distillation range and chlorine content shall be conducted on each of the individual test samples.

G-4.2 Test for the determination of the remaining characteristics, namely, refractive index, residue on evaporation, acidity and flash point shall be conducted on the composite sample as prepared under C-3.2.

G-5. CRITERIA FOR CONFORMITY

G-5.1 The lot shall be considered satisfactory in respect of the requirements tested in **G-4.1,** if each of the individual sample satisfies all the requirements.

G-5.2 The lot shall be considered satisfactory in respect of the requirements tested in G-4.2, if the test results on the composite sample satisfy the corresponding requirements.

G-5.3 The lot shall be declared to be in conformity with all the requirements of this specification, if it has been found satisfactory in G-5.1 and G-5.2.

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Printed at Delhi Printers, Delhi-6, India